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CAVITATION DAMAGE MEASUREMENTS BY RADIOTRACER ANALYSIS

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ABSTRACT

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No method exists at present for continuous measurement of the cavitation-erosion damage on metallic specimens. By using radioactive isotopes, the development of a technique that would give information not only on wear rate but on debris particle size and composition was attempted. Samples of 302 stainless steel and 1010 carbon steel were irradiated and exposed to cavitation in a venturi. Mercury and water were used as test fluids.

Meaningful data were obtained particularly with respect to particle size distribution and debris components, and their variation with particle size. In the water tests a useful measure of damage rate also was attached. The mercury tests indicated the possible technological difficulties which may be encountered in the application of this method. These are primarily concerned with the capability of the liquid to wet the debris particles and to maintain a uniform slurry.

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I. INTRODUCTION

The use of irradiated tracers for measurements of erosion and wear rates for various devices is not new, and was in fact applied previously to the measurement of cavitation damage in water[1] by the present group. In addition to providing a potentially continuous and instantaneous record of damage rate, i.e., weight or volume loss, without disassembly and examination, this technique is capable of providing information on the size spectrum and components of damage particles. A measurement of damage rate as well as of particle size distribution for stainless steel specimens submitted to cavitating water flow in a venturi was in fact achieved in the previous experiment[1]. As far as it is known, the latter measurement is still unique in the cavitation field.

The present paper describes an extension of the technique to steel specimens exposed to cavitating mercury in a venturi. Severe technological difficulties, as compared to the water experiment[1], were encountered both in maintaining a homogeneous slurry of the mercury and debris particles, and in filtering the mercury. These difficulties were primarily associated with the lack of good uniform wetting between the mercury and the debris particles. They serve to emphasize the possible difficulty of extending this apparently attractive technique to liquids whose fluid-dynamic behavior may not be easily predictable. Nevertheless, as developed later, certain significant new information did result from the mercury tests, particularly with regard to debris size spectrum and components.

II DESCRIPTION OF FACILITY

The previous experiments[1], as well as the present set, were conducted in a closed-loop facility (Figure 1) described in detail elsewhere[1,2]. It was originally operated with water and more recently with mercury. The most significant features from the viewpoint of the present experiments are summarized below.

Cavitation is caused to occur in a plexiglas venturi nominal 1/2 inch cylindrical throat diameter (Figure 2), and damage is observed on two small tapered test specimens (Figure 3), inserted parallel to the flow through the wall of the diffuser section. By suitable adjustment of the pressures and flow, the apparent termination of the vaporous cavitation region can be caused to occur approximately at the axial midpoint of the specimens, as it was in these tests, or elsewhere, if desired. This condition has been called "Standard Cavitation", and the visually apparent termination point of the cavitating region is indicated in Figure 2. Throat velocity is an independent variable which was set at $\sim$ 70 ft./sec. for the water tests[1], but at $\sim$ 34 ft./sec. for the present mercury tests, due to power and pressure limitations. For either condition the fluid transit time around the loop is of the order of a few seconds, and the Reynolds' number at all points is in the highly turbulent range. Fluid temperature in all cases was approximately ambient.

The loop (Figure 1) is powered by a vertical overhung shaft, centrifugal, sump pump (Figure 4). There is a strong

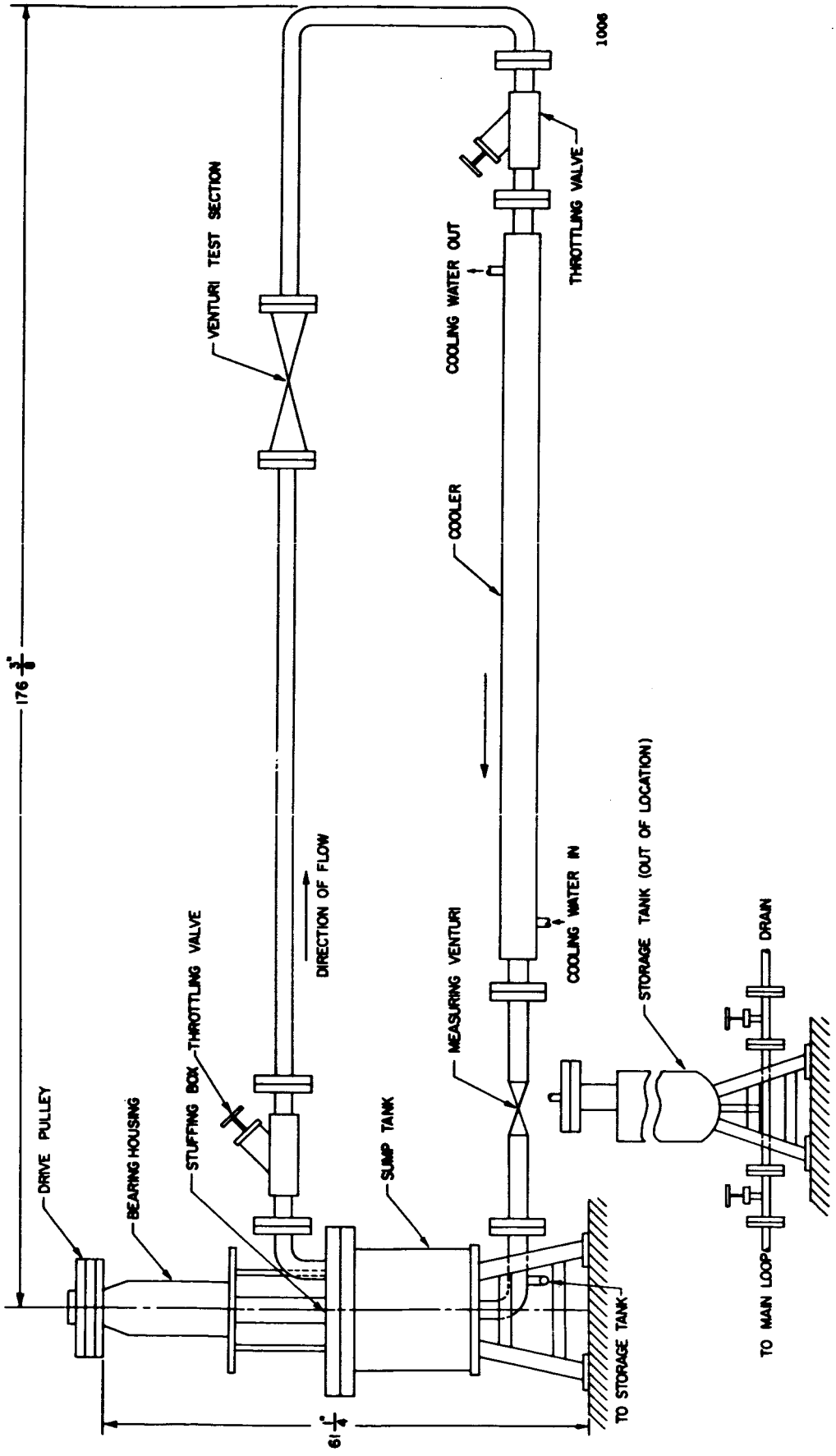

Figure 1. Sketch of Overall Liquid Metal Loop.

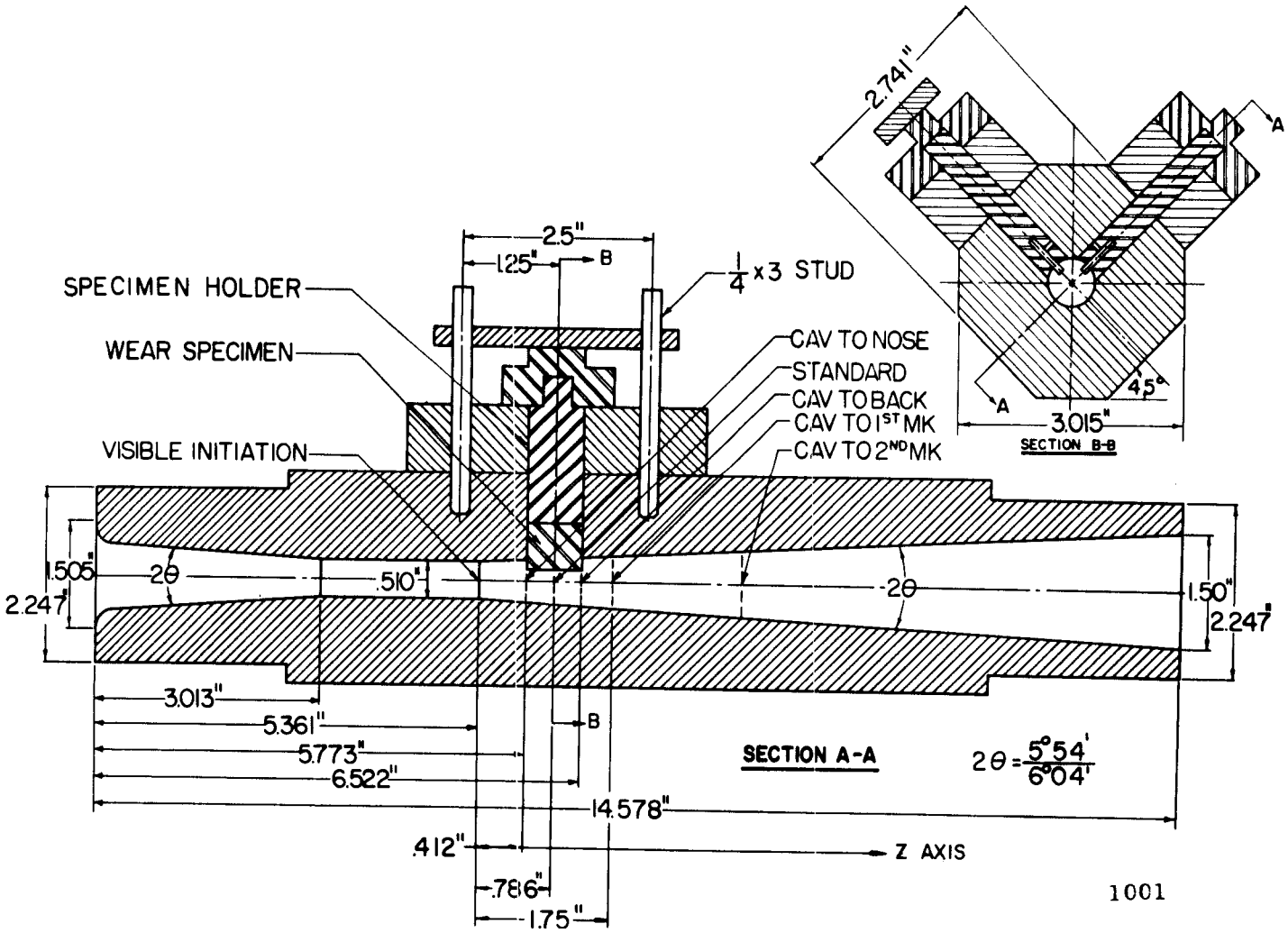

Figure 2 Drawing of the damage test venturi showing location of specimens, specimen holders, and cavitation termination points.

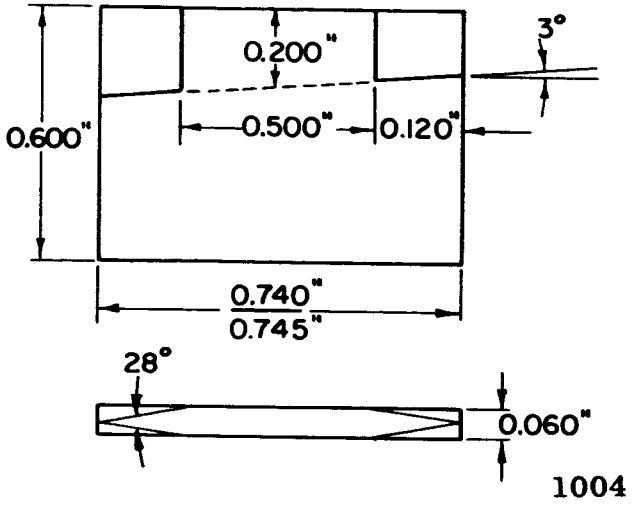

Figure 3 Drawing of damage test specimen.

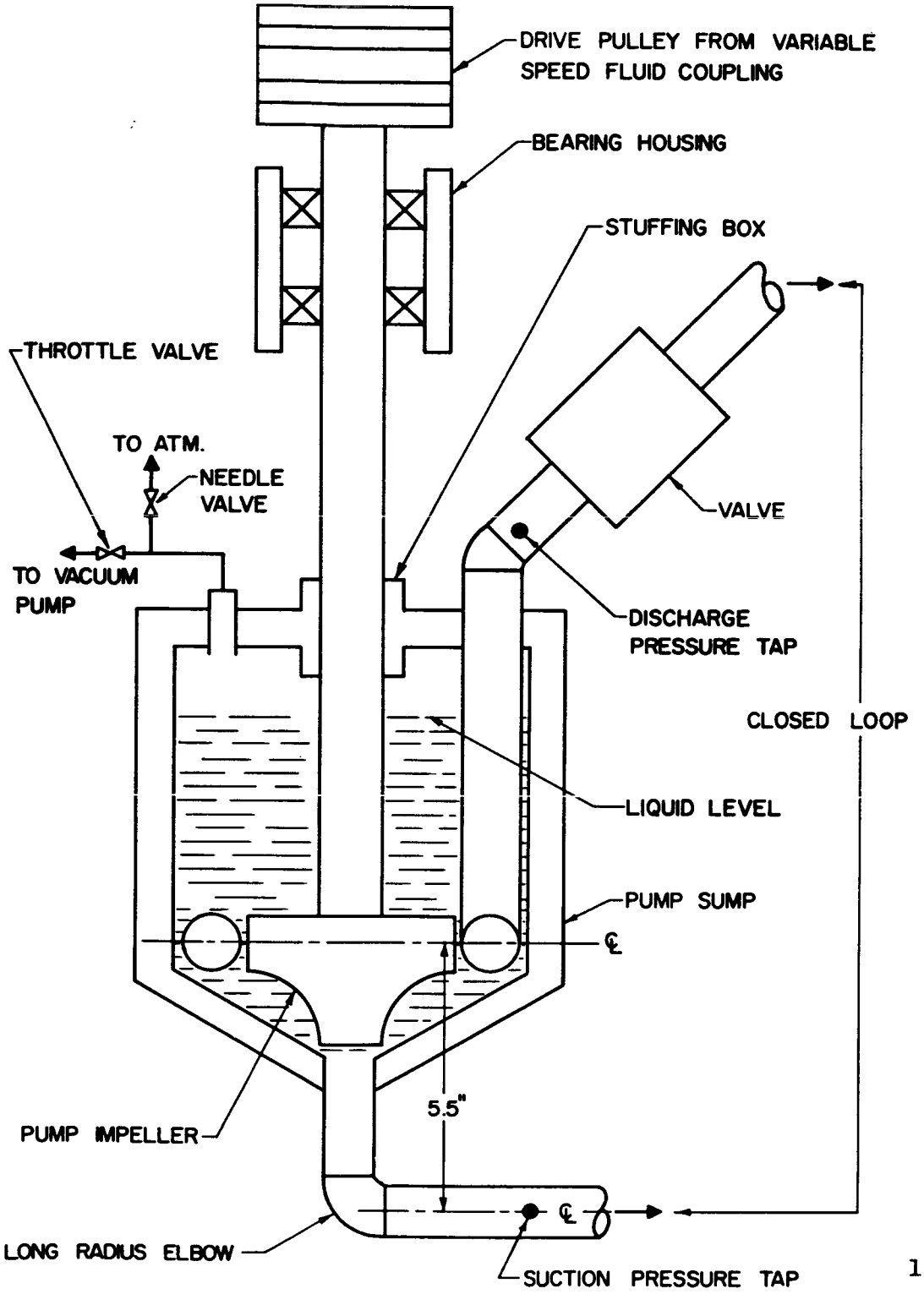

Figure 4 Schematic of pump in mercury loop.

separating effect upon foreign matter of density less than that of the fluid due to the centripetal action of the impeller, resulting in a tendency for such material to escape from the main stream into the sump, where it is trapped and floats to the surface. This effect was strongly evident during the mercury tests. Fluid circulation in the sump is very limited since the sump is closely-packed with a matrix of vertical stainless steel rods.

III EXPERIMENTAL PROCEDURE

A. Summary of Water Tests[1]

Filtering in the water tests was accomplished using a filter rack (Figure 5) accomodating four cloth or paper filters in series. The rack assembly was inserted into a small by-pass stream from the loop, activated when desired, such that all water entering the filter system passes through each filter. At the conclusion of the 20 hour test, part of the loop water was by-passed through the filter rack, while the main circulating stream was held at full velocity to maintain agitation. Two such runs were made. A final run was made with the main stream essentially stagnant to determine whether settling rates and/or entrapment were significant. The activity of the filters was determined with a 2π gas-flow proportional counter. The relationship between weight of debris and counts per minute was determined by calibration with a standard solution, obtained by dissolving in concentrated hydrocloric acid heated at 200°F, a carefully weighed piece cut from one of the radioactive specimens.

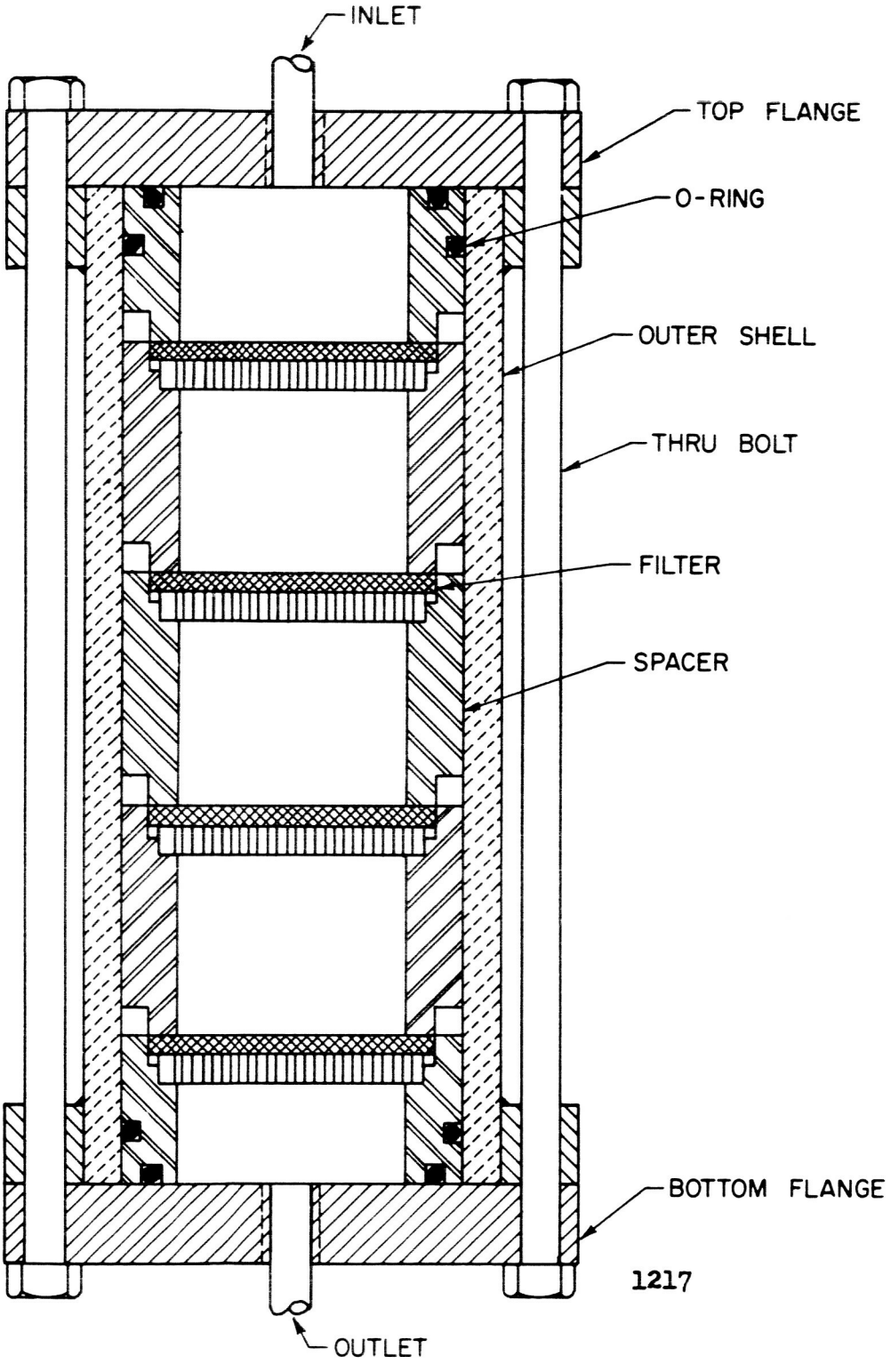

Figure 5 Drawing of the filter rack assembly.

In addition 5 cc samples were withdrawn from the loop periodically during the test. These were later evaporated on stainless steel planchets, and counted. The results obtained included a particle size measurement as well as an indication of rate of weight loss from the specimens.

B. Estimate of Required Irradiation for Mercury Tests

Mercury tests were conducted both on Type 302 stainless steel and 1010 carbon steel. Since the stainless steel test was conducted first, greater efforts were devoted to an estimate of the required irradiation in this case. The estimates were based on the following:

i) Anticipated cavitation damage-rate considering previous tests with non-irradiated specimens where weight loss had been measured directly.

ii) Minimum required specific activity in mercury for good counting statistics, assuming debris to be uniformly dispersed through the entire facility contents.

iii) Irradiation properties of Type 302 stainless steel.

iv) Substantial factors to allow for uncertainties in various estimated factors.

The irradiation was obtained by inserting the specimens in a sealed quartz tube into the University of Michigan 1 MW swimming-pool reactor. Allowance was taken for the non-continuous operating schedule of this reactor[3]. The irradiation actually attained was of course verified by direct measurement.

The maximum allowable irradiation is governed by two factors:

i) Irradiation damage to test specimen - Too high an irradiation will alter the mechanical properties of the material to be tested and thus destroy to some extent the validity of the test. Fortunately the limit for metallic materials is quite high, as compared, e.g., to plastics, other organics, etc. No measurable change in the mechanical properties of steels is expected for irradiations below about 10^{18} nvt fast flux[4,5]. The fast flux in the U-M reactor is about 5% of the thermal flux. Thus it was estimated that the stainless steel specimens in these tests had received about 1.5×10^{17} fast nvt and the carbon steel about 0.5×10^{17}. In either case the margin for a further increase in irradiation is small, so that this limitation even for metals may be significant for tests of this sort. It might very well be prohibitive for some non-metallic materials.

ii) Safe handling - In the present case no difficulty on this score was encountered. This requirement, rather than posing an absolute limitation, is a function of the equipment on hand, and in most cases can be easily met.

C. Irradiation Properties of Steels

A meaningful estimate of irradiation to be attained from a given exposure in the reactor requires a detailed knowledge of the trace components in the material to be irradiated, of their absorption cross-sections, and of the decay schemes of each. For the stainless steel a chemical analysis of a sample from the stock to be used was obtained, and from this data, a complete analysis of the nuclear properties of the different isotopes present (Table I). When all those isotopes having half-lifes

Line	Isotope	Natural Abundance	b%	W Atomic weight	N atoms/gm sample	σ_a barns	Isotope formed	Type of decay	Beta Energy Mev	Gamma Energy Mev	Half-life $T_{\frac{1}{2}}$	λ hr^{-1}
1	Fe-54	5.84	68.39	55.85		2.8	Fe-55	EC		no γ	2.94 y	
2	Fe-56	91.68				2.6	Fe-57	γ		0.014	10^{-7}s	
3	Fe-57	2.17				2.5	Fe-58	stable				
4	Fe-58	0.31			2.29×10^{19}	1.01	Fe-59	β^-, γ	.271(46%) .462(54%) 1.56(.3%)	.191(2.8%) 1.098(57%) 1.289(43%)	45.1d	6.41×10^{-4}
5	Mn-55	100	1.28	54.94	1.405×10^{20}	13.3	Mn-56	β^-, γ	2.81(50%) 1.04(30%) 0.65(20%)	.845(100%) 1.76(30 %) 2.17(20 %)	2.59h	0.27
6	Si-28	92.27	0.68	28.09		0.08	Si-29	stable				
7	Si-29	4.68				0.28	Si-30	stable				
8	Si-30	3.05				11×10^{-2}	Si-31	β^-, γ	1.471	1.26(.07%)	2.62h	
9	Cr-50	4.31	18.35	52.01	9.15×10^{19}	15.9	Cr-51	EC, γ		.325(9 %)	27.9 d	1.035×10^{-3}
10	Cr-52	83.76				0.76	Cr-53	stable				
11	Cr-53	9.55					Cr-54	stable				
12	Cr-54	2.38				0.38	Cr-55	β^-	2.85		3.6 m	
13	Ni-58	67.76	10.72	58.71		4.20	Ni-59	EC			10^5 y	
14	Ni-60	26.16					Ni-61	stable				
15	Ni-61	1.25					Ni-62	stable				
16	Ni-62	3.66				15	Ni-63	β^-	0.067			
17	Ni-64	1.16			1.277×10^{19}	1.52	Ni-65	β^-, γ	2.10(69%) 1.01(8 %) 0.60(23%)	1.49(18.8%) 1.12(12.9%) 0.37(4.9%)	2.56h	0.27
18	P-31	100	0.01	30.975		0.19	P-32	β^-	1.707		14.22d	
19	Cu-63	69.1	0.25	63.75		4.51	Cu-64	EC, β^+, β^-	.571, 0.65	1.35(.5%)	12.80h	
20	Cu-65	30.9				1.80	Cu-66	β^-, γ	several	several	5.10 m	
21	Mo-92	15.86	0.21	95.95		$<6 \times 10^{-3}$	Mo-93	EC			> 2 y	
22	Mo-94	9.12					Mo-95	stable				
23	Mo-95	15.70					Mo-96	stable				
24	Mo-96	16.50					Mo-97	stable				
25	Mo-97	9.45					Mo-98	stable				
26	Mo-98	23.75				0.51	Mo-99	β^-, γ	several	several	66 h	
27	Mo-100	9.62				0.20	Mo-101	β^-, γ	several	several	14.61m	
28	Sn-112	0.95	0.04	118.7		1.3	Sn-113	EC,		0.392	119 d	
29	Sn-114	0.65					Sn-115	stable				
30	Sn-115	0.34					Sn-116	stable				
31	Sn-116	14.24				6×10^{-3}	Sn-117	stable				
32	Sn-117	7.57					Sn-118	stable				
33	Sn-118	24.01				10×10^{-3}	Sn-119	stable				
34	Sn-119	8.58					Sn-120	stable				
35	Sn-120	32.97					Sn-121	β^-	0.383		27.5 h	
36	Sn-122	4.71					Sn-123	β^-, γ	1.260	0.153	39.5 m	
37	Sn-124	5.98				0.2	Sn-125	β^-, γ	several	several		
38	Co-59	100	0.03	58.94	3.07×10^{18}	20 ± 3	Co-60	β^-, γ	0.312	1.17, 1.33	5.24y	1.5×10^{-5}
39	Va-50	0.25	0.04	50.95			Va-51	stable				
40	Va-51	99.75				4.5	Va-52	β^-, γ	2.470	1.44	3.76 m	
	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)

TABLE I Nuclear Properties of the Isotopes Present in 302 Stainless Steel After Irradiation.

either of minutes or less or 10^2 years or more, and those which are pure beta emitters, or have a very small abundance in the sample are neglected, it is found that of the initial forty radioactive isotopes only five are significant: Fe-59, Mn-56, Cr-51, Ni-65, and Co-60. While a similarly detailed analysis was not made for the 1010 carbon steel tested later, it is likely that the same isotopes would be important due to the presence of at least trace quantities of these elements. From the information for stainless steel the required reactor exposure was calculated[6,7]. (172 hours of reactor on-time, corresponding to about 1000 hours elapsed time).

D. Stainless Steel Run

Two Type 302 annealed stainless steel specimens were used. Inadvertently a direct weight loss measurement was omitted. However, it was estimated from post-test microscopic observation, comparing with specimens for which weight loss had been obtained, that the weight loss was of the order of 2.4 mg, which is about as expected. Run duration was 26 hours, continuous.

Samples of mercury were taken periodically throughout the test both from the pump sump and from the main loop stream. It was attempted to filter these using the rack previously described (Figure 5). In some cases only one filter was used with pore size of 0.2 micron; in others, four filters in order of decreasing pore size (0.2, 0.8, 10, and 53 microns).

The filtration of mercury proved to be, in practice, quite difficult, and it is not certain that filtration was actually achieved in all attempts. It is thought that the

difficulties are due to the following:

i) Mercury does not "wet" the filter material, so that the pressure differential necessary to force it through the small pores, because of the action of surface tension, is very high. Hence it is difficult to prevent leakage around the filters or rupture of the filters.

ii) Debris particles lighter than mercury do not easily remain homogenized with the mixture, and very quickly float to the surface. For the vertical down-flow arrangement used the mercury itself may pass through the filters and leave behind the floating debris. An up-flow arrangement, for which other difficulties were anticipated, was not tried.

In the present case a certain amount of debris was left on the filters. However, it is not certain that it was primarily from the cavitation specimens.

It was planned to count the activity on the filters using a multiple channel analyser to determine both quantity of radioactive debris as a function of particle size (as had been done for the previous water tests[1]) and the components present. However, no appreciable activity (beta or gamma) was found on the filters. Further, an external check of the loop with a Geiger counter revealed no evidence of radioactivity, although the test was inconclusive because of the self-shielding effect of the mercury and the shielding of the stainless steel piping.

Finally, a 100 cc sample was withdrawn from the loop, distilled, and the residue counted. Again the results were negative. At this point there was the possibility that substantial

cavitation damage had not actually occurred; however, visual and photographic examination of the specimens showed the expected damage for this type of tests.

Some time after completion of the test, a very small amount of the radioactive specimen was counted in a multiple channel analyzer, and a differential characteristic curve obtained for future reference (Figure 6).

E. Carbon Steel Run

At the conclusion of the stainless steel test, no knowledge of the disposition of the radioactive debris, if any, existed. Consequently, it was decided to make a second run with carbon steel for which, according to the data from previous non-irradiated specimens, the damage rate should be considerably greater. The purpose of the carbon steel test was to determine the feasibility of subsequent tests of this general type, and if possible, the disposition of the radioactive debris. Thus, for this "feasibility test", the procedures used were not nearly so elaborate.

However, the specimens were carefully weighed (repeatability of measurement about $\pm$ 0.1 mg), both before and after irradiation, and after exposure to 50 hours of cavitation.* The specimens increased in weight during irradiation by about 0.7 mg each, probably due to minor corrosion. However, their weight decreased during the cavitation test by 2 to 3 mg (Table II). The irradiation achieved for these tests was such that the

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* 34 feet per second throat velocity and "Standard Cavitation".

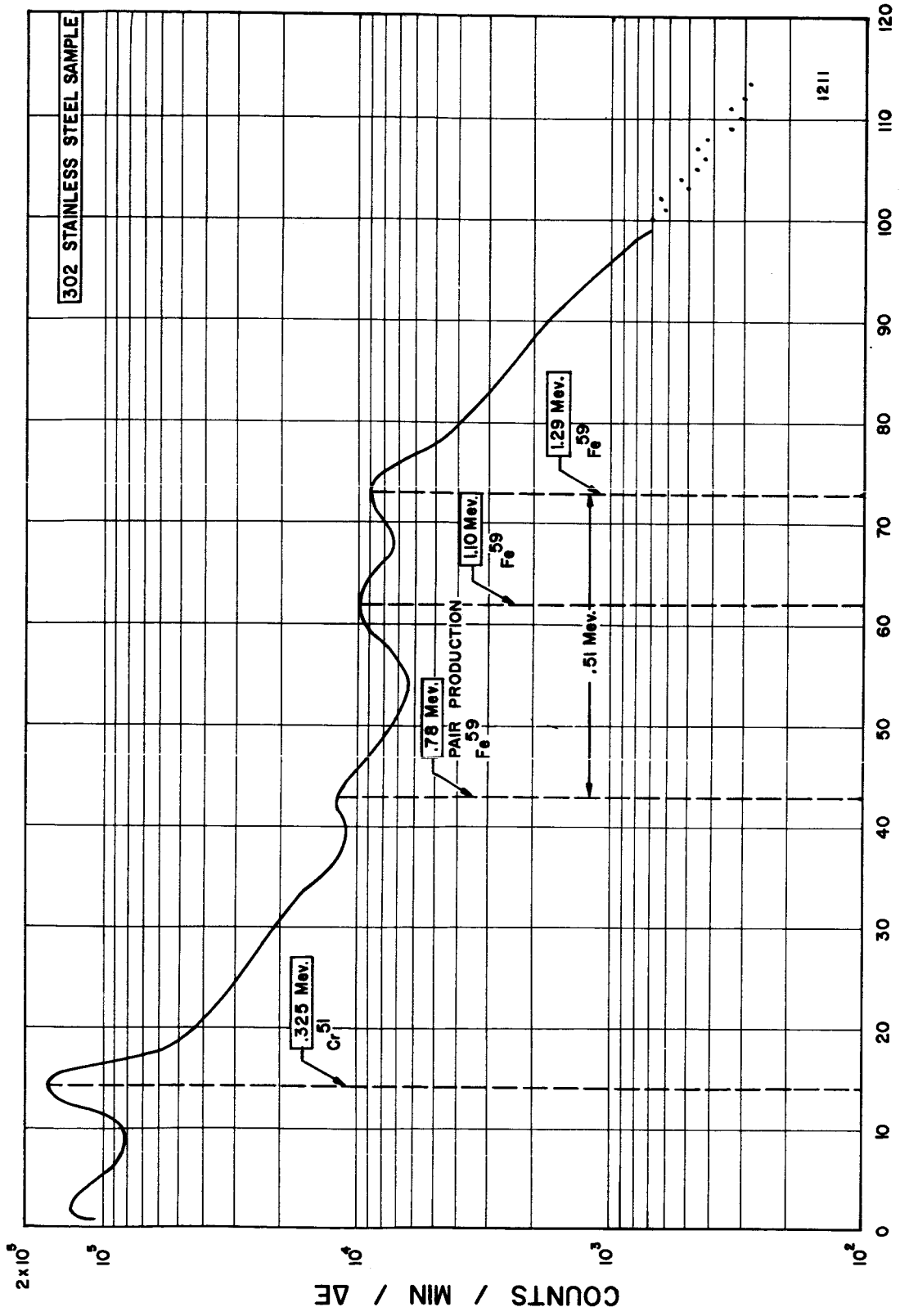

Figure 6 Differential radioactive curve for stainless steel.

TABLE II

Weights of Carbon Steel Specimens

(grams)

Date	Specimen 69-1	Specimen 70-1	Remarks
Oct. 2, 1962	3.19750	3.23504	Before irradiation.
Mar. 4, 1963	3.19830	3.23570	After reactor irradiation, but previous to cavitation run, A=20 mr/hr @ 1 ft.
Mar. 27, 1963	3.19491	3.23378	After cavitation run of 50 hours duration.
Weight Loss	0.00339	0.00192	Total weight loss for both specimens: W=5.31 mg.

contact dose rate four days after removal from the reactor was about 2 r/hr, which is about 1/2 that achieved with the stainless steel specimens.

Photomicrographs of the complete surface were taken before and after the test (Figure 7). A comparison shows that extensive damage did occur, as indicated also by the weight loss. It is probable that the damage was several times that achieved in the stainless steel test.

At the completion of the test, with the help of a Geiger counter, radioactivity was detected at two locations:

i) In the pump sump at approximately the level of the mercury surface,

ii) At flanges having vertical taps for pipe connections.

However, a 15 cc sample withdrawn from the loop again showed essentially no radioactivity.

The loop was disassembled in those regions where an external measurement had indicated radioactivity and substantial amounts of radioactive debris were recovered from two places within the sump:

i) Along the inside wall at the mercury surface, where there was a deposit of dust-like material of relatively high radioactivity,

ii) In the debris floating on the mercury.

Samples from these locations were collected on paper and burned. When the ashes were mixed with water for possible filtration, the filters immediately clogged. Consequently the ashes were calcined at 1300 ^{o}F., leaving a residue of yellowish

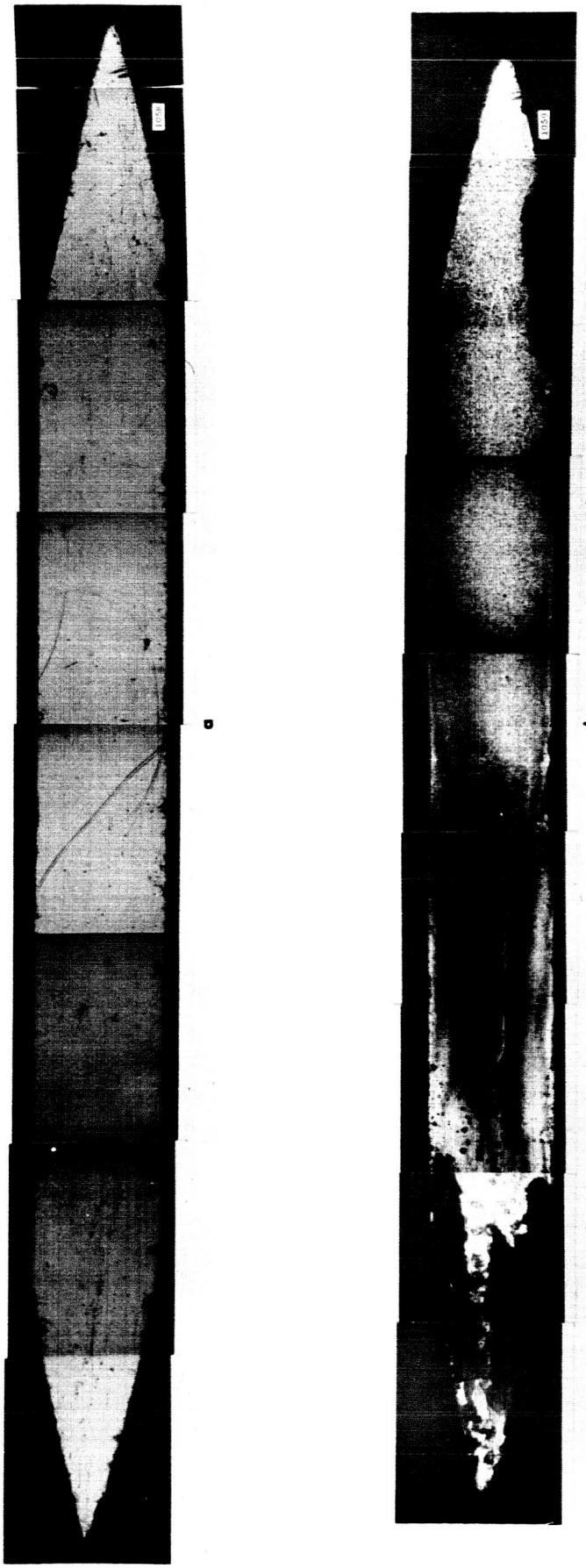

Figure 7 Photomicrograph of carbon steel specimen, (a) before irradiation and cavitation damage, (b) after irradiation and 50 hours of standard cavitation.

powder, which was then mixed with water and filtered successfully, using filter openings of 53, 10, 2, and 0.45 microns. It was assumed that the relatively high temperatures to which the material was exposed would not dimensionally affect the steel cavitation debris. The residue on the filters was then counted using a multiple channel analyser (Figure 8). Spectra substantially above background, which is included in all curves, were found for the 53 and 10 micron filters. No radioactivity was found on the 0.45 micron filter, and that on the 2 micron filter, shown in the figure, is only slightly above background.

After the test, one of the specimens was dissolved in concentrated hydrochloric acid. A standard sample of the resultant solution, suitably diluted, was counted (Figure 9). Comparison of the photopeaks of Fe-59, as well as the remainder of the spectrum for the curves shown in Figures 8 and 9 indicate that the debris components do not differ, within the accuracy of these tests, from those of the parent material, and do not depend upon particle size.

Since the material of the radioactive debris and that of the specimen are essentially the same and were irradiated at the same time, it is possible to compute the mass of material in each size range by comparing the corresponding count-rate at a given energy to the count-rate of the standard sample, at the same energy. It is of course necessary to correct the count-rate data to the same chronological time. Knowing the isotopes present, this involves no difficulty.

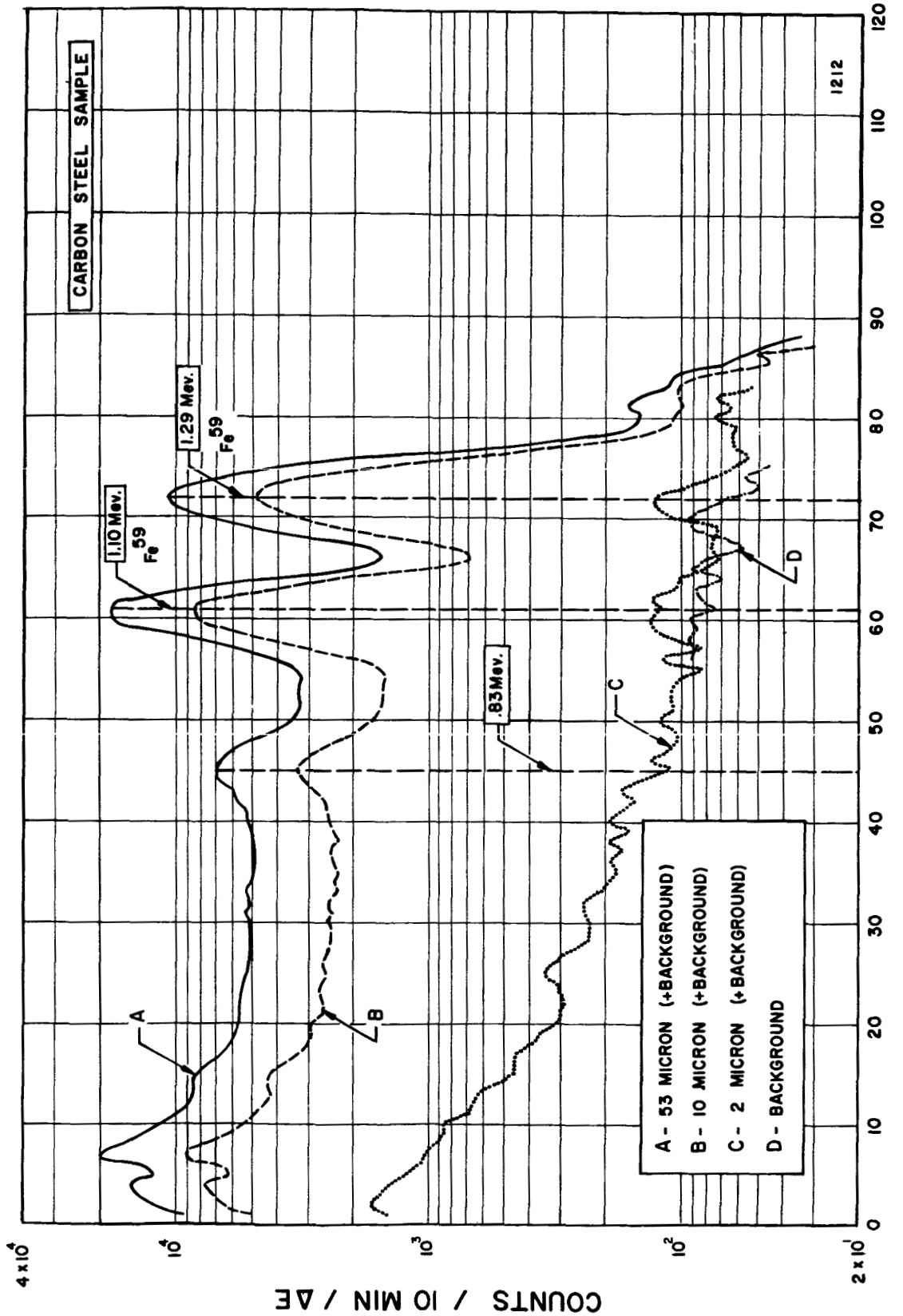

Figure 8 Differential radioactive curve of debris retained by the different pore size filters.

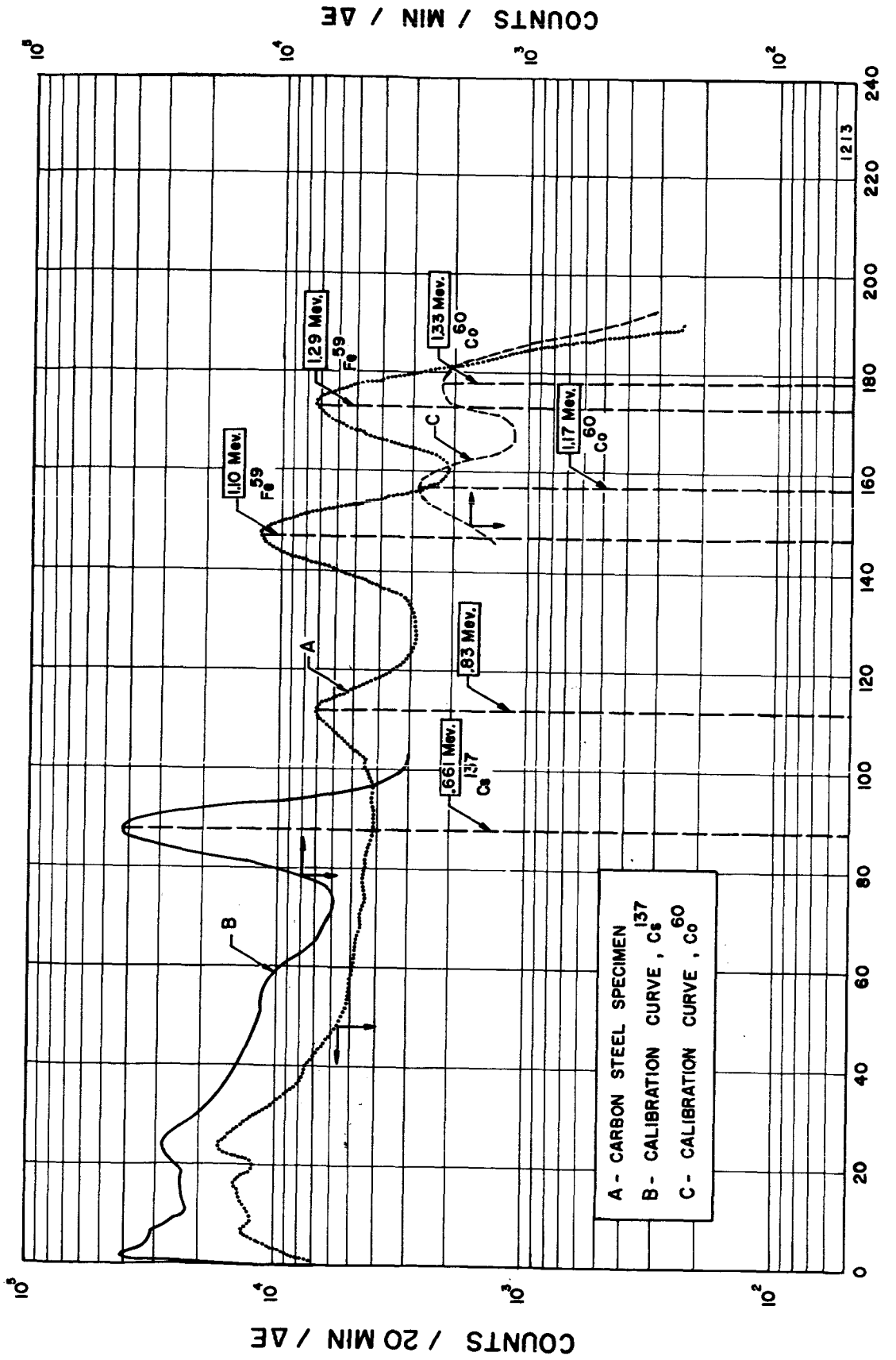

Figure 9 Differential radioactive curve of carbon steel specimen.

IV. DISCUSSION OF RESULTS

A. Mass of Debris Recovered and Particle Size Distribution

The data shown in Figures 8 and 9 was used for the calculation of the mass of material retained on the 53, 10, and 2 micron filters. The details of the calculation are given elsewhere[6,7] and hence will be merely summarized. The areas under the higher photopeak of Fe-59 (used because of its better resolution) are computed assuming a Gaussian distribution from each size-group of debris (Figure 8) and compared with the similar area from the differential curve of the standard carbon steel sample (Figure 9), corrected to the same chronological time.

The results of the calculation are summarized in Table III. The mass of the debris recovered on each filter is shown, as well as the proportion of the total measured weight loss from the test specimen which this represents. It was assumed that all the radioactive debris had originated from the carbon steel. There was a lapse of $7\frac{1}{2}$ months between the stainless steel and carbon steel experiments (sufficient for the stainless steel activity to have become negligible), and the mass of debris from the stainless steel should have been considerably smaller due to its higher resistance to cavitation-erosion and the longer duration of the carbon steel test.

Table IV also shows the number of particles in each size range, calculated assuming them to be spherical. For the largest category it was assumed that the particle diameter was equal to the specified pore size of the filter on which it was

TABLE III

Mass and Size Distribution of Particles Recovered In The Different Filters

Pore Size		Weight Recovered (mg)	% Recovered of Total Weight Loss	% Retained in Filter	% Passed Through Filter	Size of Particles	
Microns	Mils					Number	Assumed Diameter
53	2.08	.224	4.22	68.95	31.05	6000	53 μ
10	0.39	.100	1.89	30.75	.307	14750	30 μ
2	.79	.001	0.02	.307	0	18400	6 μ
Totals:		.325	6.13%				

TABLE IV

Comparison of Pits Observed With Number of Particles of Similar Size in the Debris

Size (diameter)	Counted Pits	Estimate No. of Particles
$D > 53\mu$	23	6,000
$53\mu > D > 10\mu$	1,698	14,750
$10\mu > D > 2\mu$	24,415	18,400

retained. This assumption tends to balance the following factors:

i) Some particles smaller than the pore size will be retained because of non-uniformity of pores, partial blocking by other debris, irregular shape of particles, etc., and,

ii) All particles bigger than the pore size will also be retained. However, it is doubted from visual microscopic examination of the pits, that many particles much larger than 53 microns (2.08 mils) could have existed.

For the smaller filter sizes the upper cut-off is determined by the next larger filter size. For these sizes an approximate numerical average between the limiting sizes was assumed.

Even though most of the mass was retained on the largest filter, (Table IV), the number of particles is much less than that for the 10 micron filter, which is in turn less than that for the 2 micron size. This numerical distribution is consistent in its general trend with the visual pit counts which have been performed on numerous non-irradiated specimens.

The particle size distribution, in terms of percent mass passing a given filter, is presented in Figure 10 for the carbon steel* and previous stainless steel[1] tests. It is indicated that the percent mass passing a given filter is always less for mercury than for water, i.e., the cavitation

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* The 2 micron point is not shown on this logarithmic point since the percent passing was zero.

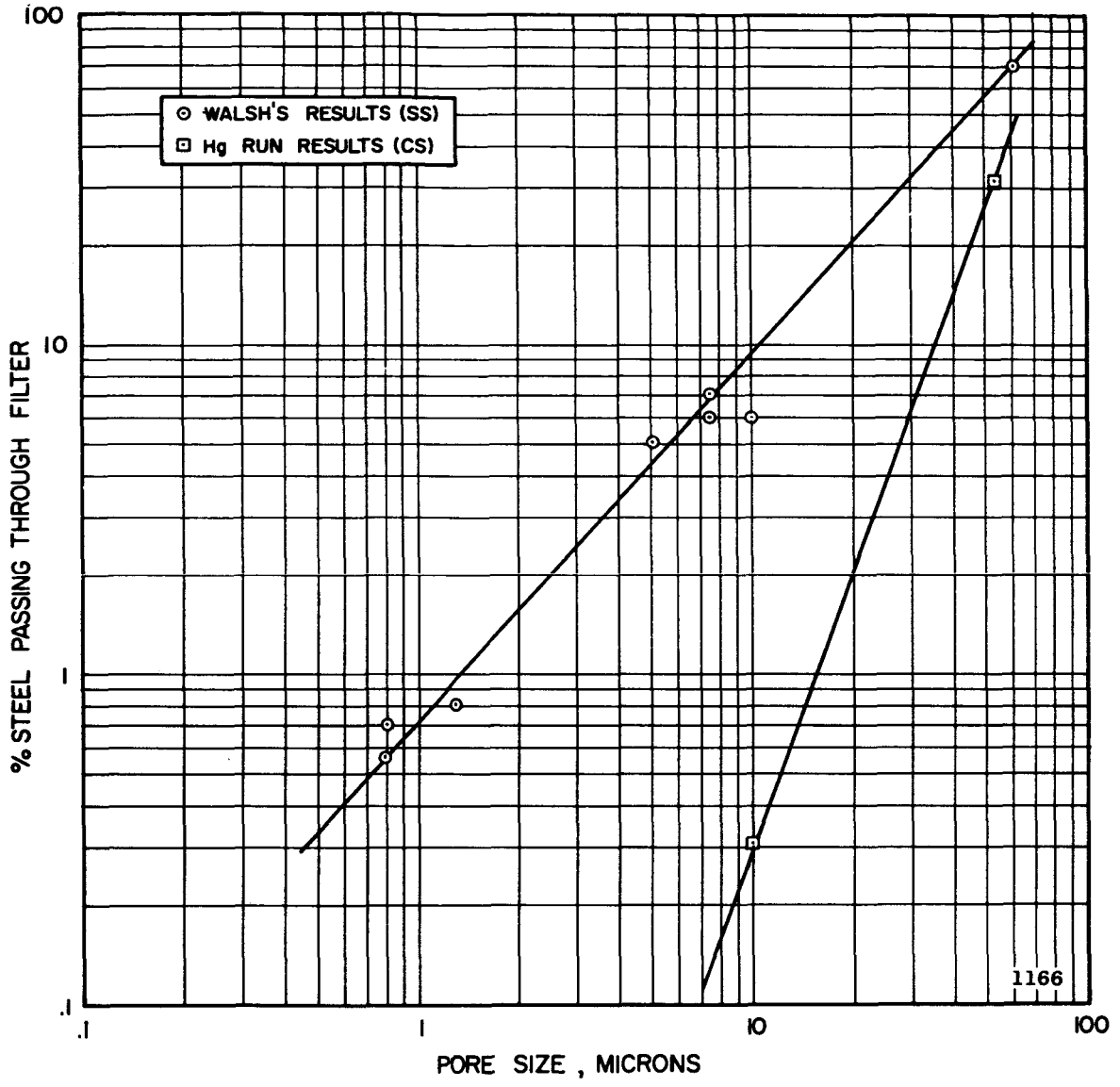

Figure 10 Percent of steel passing through filters vs. filter pore size.

debris particle sizes from the mercury test are apparently larger than for the water tests. This is not consistent with information from pit observations which show the opposite trend. The discrepancy may be due to the different filtering techniques used. However, the curves do converge towards the larger pore sizes. Thus, for either fluid most of the debris would pass an 80 micron (3 mil) filter. On the other hand, the curve indicates that only a fraction of a percent of mercury debris is of a size less than 6 to 8 microns, while about 7% by weight of the particles in water are below that size.

B. Correlation With Visual Pit Count

The composite photomicrographs of one of the carbon steel test specimens taken before and after exposure to cavitation (Figure 7) have previously been mentioned. Pit tabulation according to the filter size categories, for the regions where the pitting is not yet of the over-lapping type is compared with the estimated numbers of particles from the filter results (Table IV). The absolute numbers are not significant since the samples are not the same. However, while the ratios between different sizes agree in that the particles become more numerous as the size decreases, it is noted that this effect is much stronger for the counted pits than for the estimated numbers of particles, so that there appears to be a disproportionate number of large particles in the filtered samples which thus seem not truly representative. This is very likely to be the case since the samples were recovered from the pump sump, whence they had been centrifuged from the main stream.

The larger particles would thus be preferentially represented.

C. Components of Debris

As previously mentioned, within the precision of the data the present tests indicate no selective attack on any constituent of the carbon steel, so that the relative components of the debris are the same as those of the original specimens, independent of particle size. The curves are similar in shape and show the same peaks. Similar tests with stainless steel, where there may be a real possibility of selective attack by mercury or other liquid metals on the nickel or chromium, would be of interest for the future. The fact that the distilled sample taken after the stainless steel test showed no radioactivity does indicate no substantial solution of stainless steel in mercury for this test.

V. CONCLUSIONS

The major conclusions to be drawn involve both the feasibility of the method and various particular results from the present investigation.

A. Feasibility of Method

i) The irradiated specimen technique for cavitation-erosion studies for fluids for which disassembly and direct observation are difficult is potentially of great value. However, significant development may be required for its implementation depending upon the particular properties of the fluid-material system. Of particular importance in this respect are for the fluid: wettability, filterability, and likelihood of maintenance of homogeneous slurry

(also depending upon flow system configuration); and for the test material, susceptibility to irradiation damage and existence of suitable isotopes for irradiation. In these respects water and steel is a much more favorable combination than mercury and steel. It is believed that the alkali metals with stainless steel would prove relatively favorable because of the good wetting usually obtained.

ii) This technique represents a useful, and perhaps the only feasible method for determining particle-size distribution and relative components of the debris.

B. Particular Experimental Results

i) A particle-size distribution for cavitation damage on annealed Type 1010 carbon steel by mercury was obtained and compared with a similar measurement previously obtained for annealed Type 302 stainless steel in water[1]. The maximum particle size was about 3 mils in each case. The filtering of irradiated debris indicated that the mercury particle size range was not as great as that for water, showing fewer particles of minimum size. Since the reverse trend is indicated by visual pit counts on the damaged surfaces, it is believed that a non-representative sample was obtained for filtering. The probable explanation of this fact, involving the centrifuging action of the centrifugal pump, was previously discussed.

ii) Evidence that no selective attack occurred on the carbon steel was obtained by comparing the differential curves resulting from the debris and the original specimen. Also, within the accuracy of the data, the relative components of the

debris did not vary with particle size. A similar test for stainless steel, unfortunately not completed, would be more meaningful and perhaps instructive.

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